



MEMORANDUM

Date: April 24, 2013

To: Jessica Lepore, Project Manager, OTIE
Superfund Technical Assessment and Response Team (START) for Region 5

Prepared by: Carly Schulz, START Chemist for Region V

QA/QC Concurrence by: Renea Anglin, STAT Chemist for Region IV

Subject: Data Validation for
Vulcan-Louisville Smelting
North Chicago, IL 60064
Project TDD No. TO-02-12-06-1002

Laboratory: Test America
2417 Bond Street
University Park, IL 60484
Sample Delivery Group (SDG): 500-53164

1.0 INTRODUCTION

The START Chemist for Region 5 validated analytical data for five samples analyzed for Resource Conservation and Recovery Act-Eight (RCRA-8) metals, Volatile Organic Compounds (VOCs), Anions, Alkalinity, Total Organic Carbon (TOC), and Dissolved Gases (RSK-175). Samples were collected at the Vulcan-Louisville Smelting site on December 11, 2012. The samples were analyzed under SDG 500-53164 by Test America of University Park, IL using United States Environmental Protection Agency (U.S. EPA) methods SW846-6010B, SW846-7470A, SW846-8260B, MCAWW 300.0, SM 5310C, RSK 175, and SM 2320B.

Laboratory data were validated using guidelines set forth in the U.S. EPA Contract Laboratory Program (CLP) National Functional Guidelines (NFG)s for Superfund Organic Methods Data Review (EPA-540-R-08-01, June 2008), NFGs for Inorganic Superfund Data Review (EPA-540-R-10-011, January 2010), and applicable methodologies. The purpose of the chemical data quality evaluation process is to assess the usability of data for the project decision-making process.

Organic data validation consisted of a review of the following QC audits:

- Chain of custody (COC) and sample receipt forms review
- Sample preservation and holding time (HT)
- Blank results
- Surrogate recoveries
- Matrix spike/Matrix Spike Duplicate (MS/MSD) recovery results
- Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) recovery results
- Field Duplicates (when applicable)

Inorganic data validation consisted of a review of the following QC audits:

- COC and sample receipt forms review
- Sample preservation and HT
- Blank results
- Duplicate Sample Results
- LCS recovery results
- MS/MSD recovery results
- Field Duplicates (when applicable)

Section 2.0 of this memorandum discusses the results of organic data validation. Section 3.0 of this memorandum discusses the results of inorganic data validation. Section 4.0 of this memorandum discusses the results of the wet chemistry validation. Section 5.0 presents an overall assessment of the data. The attachment to this memorandum contains the laboratory reporting forms as well as START's handwritten data qualifications where warranted.

2.0 ORGANIC DATA VALIDATION RESULTS

The results of START's organic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted (see attachment):

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit (RL) was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives (DQO)s established for the project.

2.1 WATER SAMPLES BY 8260B – VOLATILE ORGANIC COMPOUNDS (VOCs)

2.1.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received in Chicago on ice at 1.4 °C outside of the acceptable range of 4 °C ± 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

2.1.2 SAMPLE PRESERVATION AND HOLDING TIME (HT)

The samples were analyzed on December 13, 2012. VOC samples were analyzed within holding time criteria. No discrepancies were noted.

2.1.3 METHOD BLANK (MB) RESULTS

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

One laboratory method blank sample (MB 500-172880/6) was run with this SDG. No discrepancies were noted.

2.1.4 SURROGATE (SURR) RECOVERIES

Laboratory performance on individual samples is established by means of fortifying each sample with surrogate compounds or System Monitoring Compounds (SMCs). SMCs included Dibromofluoromethane (DBFM); 1,2-Dichloroethane-d4 (DCA); Toluene-d8 (TOL); and 4-Bromofluorobenene (BFB).

Surrogate recoveries were within laboratory derived acceptable QC limits.

2.1.5 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

A MS/MSD was requested and analyzed for sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

2.1.6 LABORATORY CONTROL SAMPLE (LCS) RECOVERY RESULTS

Data for the Laboratory Control Sample (LCS) is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 500-17288/4) was fortified with the full list of VOCs and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

2.1.7 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for VOC analysis. The RPD was calculated for all detected analytes. A sample calculation using Trichloroethene (TCE) follows:

$$RPD = \frac{|170 - 160|}{[(170 + 160)/2]} * 100 = \frac{10}{165} * 100 = 6.06 \%$$

The samples were diluted due to high target analytes. Elevated reporting limits (RLs) were provided by Test America. The RPDs were calculated within acceptable QC limits. Therefore, no further action was taken.

2.1.8 GENERAL LABORATORY OBSERVATIONS

Test America diluted samples (VLF-MW-6) and (VLF-MW-6A) due to high concentrations of target analytes by factors of 10 and 20 respectively. Elevated reporting limits (RLs) were provided by Test America. Sample (VLF-MW-6A) was received by Test America with one (1) broken VOC vial. Two (2) intact VOC vials remained. Therefore, no further action was taken.

2.2 WATER SAMPLES BY METHOD RSK 175 – DISSOLVED GASES (GC VOA)

2.2.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received in Chicago on ice at 1.4 °C outside of the acceptable range of 4 °C ± 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken. The Dissolved Gas samples were shipped to Test America Burlington located in South Burlington, VT and received in Burlington on ice at 1.8° C outside of the acceptable range of 4 °C ± 2 °C on December 13, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

2.2.2 SAMPLE PRESERVATION AND HOLDING TIME (HT)

The TOC samples were analyzed on December 18, 2012 within the HT criteria for water samples. No discrepancies were noted.

2.2.3 METHOD BLANK (MB) RESULTS

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

Laboratory method blank (MB) sample (MB 200-49681/3) was run with this SDG. No discrepancies were noted.

2.2.4 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

A MS/MSD was requested and extracted from sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

2.2.5 LABORATORY CONTROL SAMPLE (LCS) RECOVERY RESULTS

Data for the Laboratory Control Sample (LCS) is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 200-49681/35) was fortified with the full list of Dissolved Gases and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

2.2.6 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for Dissolved Gas analysis. The RPD was calculated for all detected analytes within acceptable QC limits and all were within acceptable limits. Therefore, no further action was taken.

2.2.7 GENERAL LABORATORY OBSERVATIONS

Sample (VLF-MW-2) was received by Test America with one (1) broken Methane, Ethane, and Ethene (MEE) vial. Two (2) intact MEE vials remained. Therefore, no further action was taken.

3.0 INORGANIC DATA VALIDATION RESULTS

The results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted:

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The RL was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and DQOs established for the project.

3.1 WATER SAMPLES BY METHOD 6010B – RCRA-8 METALS

3.1.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received on ice at 1.4 °C outside of the acceptable range of 4 °C ± 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

3.1.2 SAMPLE PRESERVATION AND HOLDING TIME (HT)

Resource Conservation and Recovery Act Eight (RCRA-8) samples were analyzed on December 13, 2012 within the holding time criteria for water samples. No discrepancies were noted.

3.1.3 METHOD BLANK (MB) RESULTS

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities.

A laboratory method blank (MB) sample (MB 500-172801/1-A) was run with this SDG. No discrepancies were noted.

3.1.4 LABORATORY CONTROL SAMPLE (LCS) RECOVERY RESULTS

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 500-172801/2-A) was fortified with the full list of RCRA-8 metals and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

3.1.5 MS/MSD RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

A MS/MSD was requested and digested for sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

3.1.6 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for RCRA-8 metals analysis. The RPD was calculated for all detected analytes within acceptable QC limits. Therefore, no further action was taken.

3.2 WATER SAMPLES BY METHOD 7470A – MERCURY (Hg)

3.2.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received on ice at 1.4 °C outside of the acceptable range of 4 °C \pm 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

3.2.2 SAMPLE PRESERVATION AND HOLDING TIME

Mercury (Hg) samples were analyzed on December 14, 2012 within the holding time criteria for water samples. No discrepancies were noted.

3.2.3 METHOD BLANK (MB) RESULTS

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities.

Laboratory method blank (MB) sample (MB 500-172866/7-A) was run with this SDG. No discrepancies were noted.

3.2.4 LABORATORY CONTROL SAMPLE (LCS) RECOVERY RESULTS

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 500-172866/8-A) was fortified with Mercury (Hg) and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

3.2.5 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

A MS/MSD was requested and digested for sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

3.2.6 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for Mercury analysis. The RPD was calculated for all detected analytes within acceptable QC limits. Therefore, no further action was taken.

4.0 GENERAL CHEMISTRY DATA VALIDATION RESULTS

The results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted:

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

4.1 WATER SAMPLES BY METHOD SM 5310C – TOTAL ORGANIC CARBON (TOC)

4.1.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received on ice at 1.4 °C outside of the acceptable range of 4 °C ± 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

4.1.2 SAMPLE PRESERVATION AND HOLDING TIME

The TOC samples were analyzed on December 18, 2012 within the HT criteria for water samples. No discrepancies were noted.

4.1.3 METHOD BLANK (MB) RESULTS

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

Laboratory method blank (MB) sample (MB 500-1733374/3) was run with this SDG. No discrepancies were noted.

4.1.4 MATRIX SPIKE/ MATRIX SPIKE DUPLICATE (MS/MSD) RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

MS/MSDs were requested and extracted from sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

4.1.5 LABORATORY CONTROL SAMPLE (LCS) RECOVERY RESULTS

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 500-173374/4) was fortified with the full list of TOCs and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

4.1.6 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for TOC analysis. The RPD was calculated for all detected analytes within acceptable QC limits. Therefore, no further action was taken.

4.1.7 GENERAL LABORATORY OBSERVATIONS

Sample (VLF-MW-6) was received by Test America with one (1) broken TOC vial. One (1) intact TOC vial remained. Therefore, no further action was taken.

4.2 WATER SAMPLES BY METHOD SM 2320B - ALKALINITY

4.2.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received on ice at 1.4 °C outside of the acceptable range of 4 °C ± 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

4.2.2 SAMPLE PRESERVATION AND HOLDING TIME

Alkalinity samples were analyzed on December 18, 2012 within the holding time criteria. No discrepancies were noted.

4.2.3 BLANK RESULTS

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities.

Laboratory method blank sample (MB 500-173256/28) was run with this SDG. No discrepancies were noted.

4.2.4 MS/MSD RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

MS/MSDs were requested and extracted from sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

4.2.5 LABORATORY CONTROL SAMPLE (LCS) RECOVERY RESULTS

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 500-173374/4) was fortified with the full list of TOCs and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

4.2.6 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for Alkalinity analysis. The RPD was calculated for all detected analytes within acceptable QC limits. Therefore, no further action was taken.

4.3 WATER SAMPLES BY METHOD MCAWW 300.0 - ANIONS

4.3.1 SAMPLE HANDLING

Chain of custody (COC) documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact.

Water samples were collected on December 11, 2012 and were received on ice at 1.4 °C outside of the acceptable range of 4 °C ± 2 °C on December 12, 2012. Since the samples were received below the temperature range but were not frozen, no further action was taken.

4.3.2 SAMPLE PRESERVATION AND HOLDING TIME (HT)

Anion samples were analyzed on December 13, 2012. Samples VLF-MW-2 (500-53164-5), VLF-MW-5 (500-53164-4), and VLF-MW-6A (500-53164-2) were analyzed outside of HT criteria for Nitrite by approximately 10 to 40 minutes due to an instrument malfunction. Test America stated, "The problem was fixed and the samples were run in order of sampling as soon as possible." Nitrite was flagged in samples

VLF-MW-2, VLF-MW-5, VLF-MW-6A with a “J-” (estimated low) for all detected Nitrites and all undetected Nitrites with a “UJ” for undetected and estimated.

Samples VLF-MW-5 (500-53164-4) and VLF-MW-6A (500-53164-2) were analyzed outside of HT criteria for Nitrate by approximately 10 minutes to 2.5 hours due to an instrument malfunction. Test America stated, “The problem was fixed and the samples were run in order of sampling as soon as possible.” Neither sample had Nitrate detected, therefore Nitrate was flagged as UJ in samples VLF-MW-5 and VLF-MW-6A.

4.3.3 BLANK RESULTS

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities.

Laboratory method blank (MB) sample (MB 500-173019/34) was run with this SDG. No discrepancies were noted.

4.3.4 LCS RECOVERY RESULTS

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS accuracy performance is measured by %R.

LCS sample (LCS 500-173019/45) was fortified with the full list of Anions and analyzed with each batch of samples. LCS recoveries were within QC limits. No discrepancies were noted.

4.3.5 MS/MSD RECOVERY RESULTS

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

MS/MSDs were requested and analyzed for sample VLF-MW-7. MS sample (500-53164-3MS) and MSD sample (500-53164-3MSD) had percent recoveries (%Rs) and relative percent differences (RPDs) within acceptable QC limits. No discrepancies were noted.

4.3.6 FIELD DUPLICATES

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample (VLF-MW-6) had a duplicate sample collected (VLF-MW-6A) for Anion analysis. The RPD was calculated for all detected analytes within acceptable QC limits. Therefore, no further action was taken.

4.3.7 GENERAL LABORATORY OBSERVATIONS

Sample VLF-MW-2 (500-53164-5) was diluted due to an abundance of non-target analytes. Elevated Reporting Limits (RLs) were provided by Test America for this sample. No further action was taken.

5.0 OVERALL ASSESSMENT OF DATA

The analytical results meet the data quality objectives defined by the applicable method and validation guidance documentation. The analytical data is usable and acceptable as reported by the laboratory.

ATTACHMENT
SUMMARY OF VALIDATED ANALYTICAL RESULTS
AND
CHAINS-OF-CUSTODY